

Ionizing Radiation Division	43010C, 43020C	IRD-P-01
GAMMA-RAY-EMITTING RADIONUCLIDES IN SOLUTION		

Purpose

The purpose of this procedure is to describe the measurement and reporting procedures for gamma-ray-emitting radionuclides using the NIST " 4π "- γ ionization chamber.

Scope

The NIST " 4π "- γ ionization chamber (IC) is a well-type pressurized instrument which is used for measuring the radioactivity (disintegrations per second) of gamma-ray-emitting radionuclides. Scheduled calibration services for submitted samples of gamma-ray emitters are offered as a complement to the Standard Reference Material (SRM) program. These calibration services are 43010C (half-lives longer than 15 days) and 43020C (half-lives shorter than 15 days). The range of activity for the gamma-ray-emitting radionuclides is 0.4 - 60 MBq and the nominal uncertainty range is 0.47 - 2.27 percent (expanded uncertainties, $k=2$).

The IC uses calibration factors for each radionuclide previously established by direct methods of activity measurements (Hoppes 1984; NCRP 1985). Records of the direct measurements of activity used to determine k-values are kept in the K-value Record Book. For further information on specific methods refer to NIST SP-250-10. IC calibrations are checked with those of other national standardizing laboratories through intercomparisons conducted by the Bureau International des Poids et Mesures (BIPM). Results of these intercomparisons are obtainable from the BIPM. Result of NIST participation are recorded and summarized in the SIR Intercomparison Notebook.

The gamma-ray-emitting radionuclides which may be submitted to NIST for IC calibrations are those listed in the K-value Record Book.

Definitions

K-value: The relative calibration factor, K_R , is given by R_R/R , the ratio of the measured responses of the ^{226}Ra reference source and a known one bequerel of a stated radionuclide.

Equipment

Equipment manuals are maintained in 245/B47. Current instrument models and serial numbers are listed in the IC Lab Notebook. Current software and database versions are also listed in this notebook and referenced by name and file date.

Hardware

- The NIST " 4π "- γ (re-entrant) cylindrical ionization chamber (described in NIST SP 250-10)
- Multi-range electrometer
- Comparator
- Start Stop scaler
- Calendar scaler

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- Printout controller
- Thermometer/Hygrometer with computer output
- A series of 10 radium-226 reference sources (RRS)

Equipment Quality Control

Multi-range electrometer – The important characteristic of the multi-range electrometer is that the capacitor ratios be known and remain consistent. The capacitor ratios are measured approximately every two years by observing the ratios of measurements of a single RRS on multiple ranges, with the appropriate level RRS. Results are checked for consistency and recorded in the IC lab notebook.

Radium-226-reference sources - RRS ratios are checked every two years by measuring more than one RRS on the same capacitor sensitivity setting. Results are recorded in the IC Lab notebook and compared with historical data. Failure of an individual RRS would be identified as a change in ratio with two other RRSs.

Thermometer/Hygrometer – Calibration or replacement of this instrument is performed at the frequency recommended in the instrument documentation. Accuracy greater than five percent is not required to monitor unusual extremes of temperature or humidity that would affect the operation of the multi-range electrometer

Software

Iconline.exe – Iconline is a FORTRAN program running on a desktop computer. It is used to collect data from the ionization chamber, the thermometer/hygrometer, and the user. Data is stored offline for further processing

Ionol.exe – Ionol is a FORTRAN program running on a desktop computer. It is used to process the data file created by Iconline using further input from the user and information from Kvalue.dat.

Kvalue.dat – Kvalue.dat is a data file containing the most recent K-values listed in the K-value Record Book.

Validation

The validation of equipment is performed upon installation and subsequent to any change in equipment. The proper recording of data is validated by observing the results on the calendar scaler or thermometer/hygrometer and comparing this value to the value recorded by the software.

Validation of processing software is performed by manual calculation of experimental results and comparison of this value to that obtained from the program. This is performed upon the initial version and subsequent to any changes in the program.

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Results of validations are recorded in the IC Lab notebook.

Safety

Radiation safety

The room used for ionization chamber calibrations is designated as a Radiation Area. A countertop lead cave is used to house samples during measurement and to store the ^{226}Ra reference sources. In the case of high-activity sources a lead-lined “L” shield might be used while changing samples.

Procedures

Source Container and Contents

The solution to be assayed in the NIST IC should have as nearly as possible the same composition as the standards used to calibrate the chamber [4]. The gamma-ray-emitting radionuclide solution should be 5.0 ± 0.2 mL in a standard 5-mL ampoule. The specifications of these ampoules are as follows:

body diameter	16.5 ± 0.5 mm
wall thickness	0.60 ± 0.4 mm
barium content	less than 2.5 percent
lead oxide content	less than 0.02 percent
other heavy elements	trace quantities

Description of the Actual Measurement Process

The stability of this chamber, although excellent, is always monitored by measuring its response to a reference source of ^{226}Ra in equilibrium with its daughters whenever calibrations are made. The activity of the sample is taken to be proportional to the ratio of the chamber response (current) for the sample to that for the reference source. The relative calibration factor, K_R , is given by R_R/R , the ratio of the measured responses of the ^{226}Ra reference source and a known one bequerel of a stated radionuclide.

The activity, A , of the radionuclide in a sample to be measured, S , is given by $K_R R_S$, where R_S is the response (current) for the sample relative to that for the radium source. Background currents, measured just before and after counting the sample and radium, must be subtracted from the response for each. K_R diminishes from the value at the time of the original calibration with the 1600-year half life of the ^{226}Ra , and a correction for this must be made.

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Because the activity of the sample is always measured as a proportional response to a reference source of ^{226}Ra , maintenance of the standard is not dependant on the integrity of specific electronic components.

Preliminary

- Customer contact - give specifications for chemical composition and activity limits, source must be provided as 5-mL solution in an NIST-type 5-mL flame-sealed ampoule.
- NIST paperwork and acceptance procedure- submit completed NIST 364 for approval before arrival of materials, notify the Chief of Health Physics of the arrival and departure of all plutonium and uranium, complete other necessary forms (NIST 64, 77, 796A).
- Test folders - request test folder on receipt of material to be calibrated. Note dates of material received and returned.
- Run program KVUPD or examine database to determine appropriate electrometer range and ^{226}Ra reference source to be used for measurement.
- Schedule time to use the IC by blocking and initialing the required amount of time on the calendar in B47. Possible conflicts should be resolved in person with other users. Generally shorter half-life sources take precedence.
- Deliver source, or a related sample, to the appropriate person to be measured on a calibrated germanium spectrometry system according to instructions for an impurity measurement. The results and source are delivered to the analyst at the completion of this measurement.

Receiving Instructions for Radioactive Solution Samples

All radioactive sources arriving at NIST are delivered to the Health Physics Group where they are examined to determine the radiation level and possible contamination of containers and packaging.

After Health Physics releases the source to the analyst, the analyst in turn should verify that the correct radionuclide has been shipped in the activity range which has been previously discussed with the customer. Quality control procedures that should be implemented by the analyst are as follows:

1. The analyst should re-examine the source for pin holes of any type and check the ampoule seal.
2. The analyst should inspect the label attached to the primary container. This helps to insure that it is the required radionuclide and the activity amount is correct.

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3. Technical data sheets and package inserts accompanying the radionuclide package should be read carefully for special instructions on stability, quality inspection, and chemistry of the product. This information should conform to the calibration guidelines specifying the physical and chemical properties of the submitted sample solution.

Providing that no defects are observed, the sample may be transported for measurement in the NIST "4 π "- γ ionization chamber.

Step-by-Step Operating Instructions for the NIST "4 π "- γ Ionization Chamber

Sign in using the IC Log Book.

Starting of the Ionization Chamber System

The system is started from the attached computer using the following procedure:

1. Check schedule and observe system to verify it is not currently in use.
2. Verify computer system time. If necessary, correct the time using NIST network time.
3. Click on desktop icon "iconline." The system is started.
4. Answer Y or y for default values, N or n to change and supply answers. Generally use default values. Press return after each answer.

Questions are as follows:

New Bkg-O; Skip 1

BKGS=-1

Time: Day#

Input Start of Calendar Scalar HR, MIN, SEC

Reset clock on clock scalar

Computer Time: HR: MIN: SEC

Statement of agreement between computer and calendar clock

Answer Y or y if OK, N or n to reenter cal. start time

Enter Sample #: O-BKG, 999-DONE, -999 change CAPR, PTS.

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Setting the Electrometer

The coulomb dial on the electrometer must be set to 10^{-9} for all measurements.

Set the sensitivity dial to the proper setting. The range of sensitivity is 1 to 1000. The sensitivity setting for background is 1. Refer to Table 6 for determining the 1-1000 range settings.

Capacitor Ratio	Radium Reference Source*
1	Background
10	3, 10, 20
100	20, 50, 100, 200
1000	200, 500, 1000, 5000

Table 1 Sensitivity Range Settings

*The number of the radium reference source used is approximately equivalent to the number of micrograms of radium per source.

The capacitor ratio and the ^{226}Ra reference source to be used are determined based on the activity provided by the supplier when the sources are submitted, with appropriate corrections for decay.

Background Measurements

Background measurements are made initially and should be examined before continuing. If the background is inconsistent, or abnormally different, constant repetitive measurements may be necessary.

Measuring Reference Source

- 1) Place the ^{226}Ra reference source into the rigid, tight-fitting Lucite source holder and lower into the chamber. Align the markings on the source holder with those on the instrument shielding.
- 2) Change the timer controller switch from the manual to the automatic mode. Press Start. (This step is repeated each time a radium reference source or solution sample is measured.)
- 3) After the designated number of measurements has been made on the ^{226}Ra reference source, or on a sample, press the F10 key. This is done at the end of every set of measurements. The program will display the average count time and standard deviation. The sample number will be requested for the next sample which is to be counted.
- 4) Remove the holder from the chamber and replace the ^{226}Ra reference source with the sample which is to be assayed.

Measuring the Sample

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When placing the solution sample in the chamber, observe the ampoule to make certain that no liquid is in the neck of the ampoule. The sample is then counted the same number of times in each set of measurements as the radium reference source. At least four sets of 10 measurements should be made on the sample, with an equivalent number of measurements made on the radium reference source.

After all measurements have been completed on the submitted sample and the radium reference source, it is recommended that background measurements be repeated.

Data Collection

The data collection is under control of Iconline.exe. The start of the calendar scalar and other default parameters are entered as prompted, by the operator. The operator enters the sample number, number of data points to be collected, and capacitor ratio. The program obtains the current temperature and humidity from the connected instrument, records the values, and determines whether the results are within specifications for proper operation of the electrometer. If results are not acceptable the program will notify the user and will not proceed. All data is collected and stored to a disk for off-line analysis.

Measurement of impurities

Impurity-check measurements of radioactive sources to be calibrated by the ionization chamber are performed using the IRD Gamma-Ray Spectrometry System located in Building 245, Room C25 (basement). This system consists of four High Purity Germanium (HPGe) detectors and one Silicon-drifted detector. These detectors are calibrated using NIST Standard Reference Materials (SRMs), which are, in turn, validated through intercomparisons with other National Metrology Institutions and the BIPM, and the periodic submission of measured sources to the International Reference System (SIR) for gamma-ray emitting sources at the BIPM (<http://www.bipm.org/en/scientific/ionizing/radionuclides/sir/>). The calibration of each of the five detectors is done through the development of efficiency curves for different source geometries and various source-to-detector distances. These efficiency curves are verified monthly or more frequently if needed (such as when there has been a change in the instrument configuration), using previously calibrated ^{57}Co and ^{60}Co point sources to determine the efficiency value and energy calibration reproducibility. Efficiency curves for point sources (solids), 5 mL ampoules (liquids) and 33 cm³ spheres (gas) for gamma-ray emitting sources for the energy range $14 \text{ keV} < E < 3.6 \text{ MeV}$ are available for impurity and activity calibration measurements.

Gamma-ray spectra are acquired using GammaVision™ (version 2.0), a commercially available software developed and marketed by EG & G ORTEC. Data analysis is performed using Genie 2k from Canberra (operational manual located in laboratory). The choice of data analysis software was based on in-house comparisons of the results obtained by several (3) different software packages and by hand. In addition, the data analysis software used by customers was taken into account. Such commercial products are identified in order to specify these procedures

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accurately. The IRD does not recommend or endorse any commercial product, nor intend to imply that these products are necessarily the best available for the purposes of these procedures.

The uncertainty on the activity values for the impurities and/or source measurement is obtained by the expanded uncertainty as given by

$$\sigma_{imp/source} = 3\sqrt{\sigma_{eff}^2 + \sigma_{measure}^2}$$

where σ_{eff} is the uncertainty of the efficiency for the measured gamma-ray energies for the impurity and/or source, and $\sigma_{measure}$ is the uncertainty calculated by Genie 2k for the value of the activity of the measured impurity and/or source. As impurity levels are generally much lower than the nuclide being calibrated, the uncertainty on the impurity measurement is often on the order of 5% to 20%.

Processing the Data

The data processing is done using Ionol.exe. The program averages the data for each sample and corrects them for background and radioactive decay (using the established half life). The sample activity is calculated using the K-value for that radium reference source producing a current approximating that of the sample. The results, with intermediate averages if desired, are printed out.

Correction for impurities is handled by the processing program. Results of impurities are entered as an activity ratio relative to the main radionuclide and a measurement time.

Acceptance Criteria

The data is examined to see if the results show the expected activity and the standard deviation of repeated measurement on the source is checked to see if it is in the range expected for the radionuclide, normally less than 0.1 percent at 1 sigma. If there is a discrepancy in activity the source may be measured in a commercially re-entrant ionization chamber, or “dose calibrator,” as a confirmatory measurement. This measurement is performed by placing the sample in the dose calibrator source holder and entering the correct instrument setting for the radionuclide in question. Settings are found on a list attached to the instrument or in the instrument manual. If the standard deviation is abnormally high the data is examined for possible trends. The customer is contacted if a discrepancy cannot be resolved.

Calibration Report

At the end of a calibration exercise, the customer will be given a Report of Calibration stating primarily:

a) the principal radionuclide

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- b) reference time and date
- c) method of calibration
- d) certified values for activity
- e) decay-scheme assumptions
- f) assessment of radionuclidic purity
- g) overall uncertainty determinations for calibration

Reported values are checked against the data on the IC printout and this check is noted by initialing the printout. Drafts of reports are stamped as such and identified by draft number. Results of draft proofreading are noted and initialed on the draft.

Signatures are obtained at the appropriate level and the original report is embossed with a Department of Commerce seal. A copy marked file copy and two marked copies are maintained with the calibration printouts, stored in folders identified by company, radionuclide, and test number. The original report is sent to the customer.

Customer Sources

When the calibration service is completed, the technical users are notified, and the calibrated sample returned, if desired by customer. Short-lived radionuclide samples are usually measured by the customer only before transmittal to NIST, for the activity would be too low for measurement after the NIST calibration.

Uncertainty Analysis

The bases for the determination of uncertainties are the ISO Guide to the Expression of Uncertainty in Measurement and the Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results.

The purpose of this section is to explain the derivation of the various components of uncertainty. Measurements performed with the "4 π "- γ ionization chamber (IC) must be done under completely fixed conditions. This requires a stable background, no saturation losses due to incomplete charge collection because of recombination at high rates, and corrections for all impurities. Ampoules must be of the standard 5mL type, the volume in ampoules must be 5.0 ± 0.2 mL, and the ^{226}Ra reference source must be corrected for decay.

Uncertainty in activity measurements may be increased if the contribution of the background is variable, large, or both. The uncertainty quoted is derived from measurement of the variations. The operator of the chamber should make frequent measurements of the background and look for the source of any unusual variations observed.

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- K-value (Type A and Type B) – uncertainty in the determination of the k-value, including the measurement of activity by primary methods and measurements to transfer that calibration to the ionization chamber (specific uncertainties for individual k-values are listed in the K-value Record Book)
- RRS ratio (Type A) - uncertainty in the ratio of radium reference sources, when the radium source used in the measurement is different from the radium source used for the determination of the K-value
- Half life (Type A) – uncertainty in the half-life value used for decay corrections
- N measurements on this sample (Type A) – standard deviation determined from n measurements of activity ratios
- Impurities in this sample (Type A and Type B) - Radionuclidic impurities may or may not have a significant effect, depending on the radiations of the principal and impurity radionuclide and the amount of impurity present. If their half-lives are relatively short, measurements at different times may be used to check consistency. Germanium detector systems are used to identify and quantify the significant radionuclidic impurities that may be present. The uncertainty in these measurements is propagated through the calculated effect on the IC results.
- Gravimetric (Type B) – typical uncertainties from gravimetric transfers of solution, if necessary

Records

Calibration printouts and report copies are stored in folders identified by company, radionuclide, and test number.

If the source is returned, the date that the source is shipped back to the customer should be entered into the folder.

Folders of calibration records are maintained in 245/C136 for at least five years.

Documentation

K-Value Record Book – maintained in 245/E102

Ionization Chamber Laboratory Notebook – maintained in 245/B47

Ionization Chamber Log Book – maintained in 245/B47

SIR Intercomparison Notebook – maintained in 245/E108

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References

Hoppes, D. D., Basic Radionuclide Measurements at the United States National Bureau of Standards, Environmental Int. 10, 99-107 (1984).

NCRP Report 58, A Handbook of Radioactivity Measurements Procedures, National Council on Radiation Protection and Measurement, Bethesda, Maryland, (1985).

Taylor, B.N., and Kuyatt, C.E. "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results", NIST Technical Note 1297(1994).

Calhoun, J.M., Radioactivity Calibrations With the NIST "4 π " Gamma Ionization Chamber, NIST Special Publication 250-10, 1987

Filing and Retention

Calibration printouts are stored in folders identified by company, radionuclide, and test number.

If the source is returned, the date that the source is shipped back to the customer should be entered into the folder.

For customer calibration, prepare calibration report and obtain required signatures. Make copy for customer file and send original.

The IRD Quality Manager shall maintain the original and all past versions of this IRD Procedure. Copies of the current revision of this Procedure shall be placed in controlled Quality Manuals. Electronic copies of this Procedure are uncontrolled versions.

All deleted Procedures (including old revisions) shall be maintained by the IRD Quality Manager. All old revisions shall be maintained until such time as it is decided to delete the Procedure. Once the decision has been made to delete the Procedure, only the last revision shall be maintained by the IRD Quality Manager.

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Appendix A. Sample Calibration Report



U.S. DEPARTMENT OF COMMERCE
National Institute of Standards & Technology
Gaithersburg, MD 20899

REPORT OF CALIBRATION

for

COMPANY, INCORPORATED
TOWNESVILLE, STATE

Radionuclide	Linolium-100
Source identification	1234-abc
Source description	Liquid in a NIST borosilicate-glass flame-sealed 5-mL ampoule ^{(1)*}
Solution composition	LoCl ₂ in 0.1 M HCl ⁽²⁾
Activity	1.235 x 10 ⁵ Bq
Relative expanded uncertainty	1.5 percent ⁽³⁾
Reference time	1200 EST 30 February 2004
Photon-emitting impurities (Activity ratios at reference time)	¹⁰¹ No/ ¹⁰⁰ Lo: (1.2 ± 0.3) x 10 ⁻⁵ ⁽⁴⁾
Half life	123.45 ± 0.06 days ⁽⁵⁾
Measuring instrument	NIST "4π"-γ ionization chamber calibrated by apple π(e+x)-γ coincidence counting
Measurements Performed by	<i>Signature here</i> Jeffrey T. Cessna, Physicist

Signature here

Lisa R. Karam, Acting Division Chief
Ionizing Radiation Division
Physics Laboratory

For the Director,
National Institute of Standards and Technology by

Signature here

Michael P. Unterweger, Acting Group Leader
Radioactivity Group
Physics Laboratory

Gaithersburg, MD 20899
Report Issued: 1 March 2004
NIST Folder No.: xxxxx-xx

*Notes on back

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NOTES

- (1) The solution was prepared by Company, Incorporated (CI), Townesville, State. CI reported the mass to be 4.9938 grams.
- (2) Description provided by CI.
- (3) The uncertainty analysis methodology and nomenclature used for the reported uncertainties are based on uniform NIST guidelines and are compatible with those adopted by the principal international metrology standardization bodies [cf., B.N. Taylor and C.E. Kuyatt, *NIST Technical Note 1297* (1994)].

The relative combined standard uncertainties, $u_c = 0.75$ percent, is the quadratic combination of the standard deviations (or standard deviations of the mean where appropriate), or approximations thereof, for the following component uncertainties:

a)	40 ionization-chamber measurements on this sample	0.02 percent
b)	k - value	0.72 percent
c)	photon-emitting impurities in this sample	0.02 percent
d)	radium reference sources ratio	0.08 percent
e)	radium reference source positioning	0.10 percent

The relative expanded uncertainty, $U = 1.5$ percent, is obtained by multiplying u_c by a coverage factor of $k = 2$ and is assumed to provided an uncertainty interval of approximately 95 percent confidence.

- (4) Limits of detection for impurity gamma rays are:

$$\begin{aligned}
 &1.2 \times 10^5 \gamma \cdot s^{-1} \text{ between 30 and 110 keV,} \\
 &3.8 \times 10^4 \gamma \cdot s^{-1} \text{ percent between 130 and 350 keV, and} \\
 &4.6 \times 10^3 \gamma \cdot s^{-1} \text{ percent between 400 and 1800 keV,}
 \end{aligned}$$

provided that impurity photons are separated in energy by five keV or more from those emitted in the decay of limolium-100. Limits are as of impurity measurement time, 32 January 2004.

- (5) Evaluated Nuclear Structure Data File (ENSDF), February 2004.

For further information, please contact Jeffrey T. Cessna at (301) 975-5539.

Source identification 1234-abc
NIST Folder No. xxxxxx-xx

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